

# Document made available under the Patent Cooperation Treaty (PCT)

International application number: PCT/GB05/050171

International filing date: 30 September 2005 (30.09.2005)

Document type: Certified copy of priority document

Document details: Country/Office: EP  
Number: 04256077.1  
Filing date: 01 October 2004 (01.10.2004)

Date of receipt at the International Bureau: 25 November 2005 (25.11.2005)

Remark: Priority document submitted or transmitted to the International Bureau in compliance with Rule 17.1(a) or (b)



World Intellectual Property Organization (WIPO) - Geneva, Switzerland  
Organisation Mondiale de la Propriété Intellectuelle (OMPI) - Genève, Suisse



Europäisches  
Patentamt

European  
Patent Office

Office européen  
des brevets

Bescheinigung

Certificate

Attestation

Die angehefteten Unterlagen stimmen mit der ursprünglich eingereichten Fassung der auf dem nächsten Blatt bezeichneten europäischen Patentanmeldung überein.

The attached documents are exact copies of the European patent application described on the following page, as originally filed.

Les documents fixés à cette attestation sont conformes à la version initialement déposée de la demande de brevet européen spécifiée à la page suivante.

Patentanmeldung Nr. Patent application No. Demande de brevet n°

04256077.1

Der Präsident des Europäischen Patentamts;  
Im Auftrag

For the President of the European Patent Office

Le Président de l'Office européen des brevets  
p.o.

R C van Dijk





Anmeldung Nr:  
Application no.: 04256077.1  
Demande no:

Anmeldetag:  
Date of filing: 01.10.04  
Date de dépôt:

Anmelder/Applicant(s)/Demandeur(s):

BAE Systems PLC  
6 Carlton Gardens  
London SW1Y 5AD  
GRANDE BRETAGNE

Bezeichnung der Erfindung/Title of the invention/Titre de l'invention:  
(Falls die Bezeichnung der Erfindung nicht angegeben ist, siehe Beschreibung.  
If no title is shown please refer to the description.  
Si aucun titre n'est indiqué se référer à la description.)

High-emissivity radiator

In Anspruch genommene Priorität(en) / Priority(ies) claimed / Priorité(s)  
revendiquée(s)  
Staat/Tag/Aktenzeichen/State/Date/File no./Pays/Date/Numéro de dépôt:

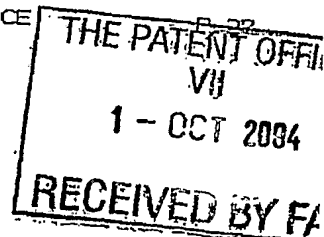
Internationale Patentklassifikation/International Patent Classification/  
Classification internationale des brevets:

F28F/

Am Anmeldetag benannte Vertragsstaaten/Contracting states designated at date of  
filing/Etats contractants désignées lors du dépôt:

AT BE BG CH CY CZ DE DK EE ES FI FR GB GR HU IE IT LU MC NL  
PL PT RO SE SI SK TR LI





- 1 -

## HIGH-EMISSIVITY RADIATOR

This invention relates to a radiator and to a method of making a radiator. In particular, this invention relates to a radiator having thin-film coatings that serve to increase the emissivity of the entire structure thereby better to match  
5 the thermal radiation of a "black-body" radiator. The invention also relates to how such a radiator is made.

All bodies will generally emit thermal radiation, and the efficiency at which they emit thermal radiation is defined as that body's emissivity. The emissivity is quantified as a percentage of the thermal radiation from an ideal  
10 source, commonly referred to as a black body. Although it is theoretically impossible to realise the perfect black body, current optimised optical designs in combination with careful selection of materials have seen radiators with emissivity values as high as 95% over their intended operating wavelength range. As is well known, emissivity will vary over the electromagnetic spectrum  
15 and so is optimised for a particular operational wavelength range.

Radiators with high emissivity are useful in a number of industrial and research applications. For example, they may be used as calibration standards for pyrometers or may be used in a thermal picture synthesiser to form infra-red images for the testing of infra-red imaging or targeting equipment.

20 Current designs are often based on optical stacks and are multilayers. This multilayer approach needs highly accurate thickness control of each layer otherwise the performance of the entire structure is compromised. Single layer emitters are relatively thick and have inferior mechanical properties. These high-emissivity radiators have been bulky. The increased mass results in a  
25 slower response time to reach their operating temperature and hence to produce the desired thermal radiation spectrum. This is a particular problem in thermal picture synthesising where imaging must be performed at typical television frame rates (e.g. 50 Hz). In addition, the bulk of the high-emissivity radiators means that they cannot be applied to an object without the size and,  
30 possibly, shape of that object changing significantly.

- 2 -

Against this background, and from a first aspect, the present invention resides in a radiator comprising a substrate, an amorphous carbon layer and a metallic carbide-forming layer interposed between the substrate and amorphous carbon layer. This arrangement allows thin-films of the metal and amorphous carbon to be deposited that have very little bulk and hence mass. In addition, the thin films will conform to the shape of an object to be treated without substantially altering the size or shape of that object. Moreover, provision of the metallic and amorphous carbon layers produces a radiator that can match the 95% emissivity of known designs.

10 Preferably, the amorphous carbon layer has a thickness in the range 0.1 to 1.0 micrometres, a thickness in the range 0.3 to 0.6 micrometres being particularly preferred and a value of substantially 0.4 micrometres being preferred still further. These values have been found to produce high emissivity in the infra red region.

15 The carbide-forming layer may be integral with the substrate or it may be separate from the substrate. For example, if the substrate itself is capable of forming a carbide layer when amorphous carbon is deposited thereon, there is no need to deposit a separate metallic carbide-forming layer. On the other hand, the substrate may not form a carbide or may form a carbide only poorly, in which case it is either necessary to deposit a metallic carbide-forming layer or better results may be obtained by depositing a separate metallic carbide-forming layer. For example, the metallic carbide-forming layer may comprise titanium. This arrangement leads to a titanium layer adjacent an amorphous carbon layer, the reaction between these layers forming titanium carbide at their interface. The titanium may be the substrate material or may, for example, be a layer deposited onto a semiconductor substrate. Optionally, the titanium layer has a thickness in the range of 0.1 micrometres to 1.0 micrometres, a thickness of substantially 0.3 micrometres being particularly preferred.

25 Optionally, the amorphous carbon layer and/or the metallic carbide forming layer is formed by sputter deposition.

- 3 -

The amorphous carbon layer may, optionally, be protected by a protective layer. This may then afford protection from damage by abrasives and/or oxidation. To avoid attenuation of infra-red thermal radiation emitted by the radiator, the protective layer should be substantially transparent to infrared radiation. For example, the protective layer may optionally comprise SiC, SiO<sub>2</sub>, diamond or diamond-like carbons.

From a second aspect, the present invention resides in a method of making a radiator comprising the steps of forming a metallic carbide-forming layer on a substrate and forming an amorphous carbon layer on the metallic carbide-forming layer. The metallic carbide-forming layer and the amorphous carbon layer may be formed in any order. For example, the metallic carbide-forming layer may be deposited onto a substrate and the amorphous carbon layer subsequently deposited onto the metallic carbide-forming layer. Alternatively, the amorphous carbon layer may be deposited directly onto a substrate if the substrate is capable of forming a metallic carbide itself: in this way, deposition of the amorphous carbon may cause a reaction with the substrate thereby forming a carbide layer automatically. Such a radiator will have the advantages discussed above in relation to the first aspect of the present invention.

Preferably, the amorphous carbon layer is formed to a depth in the range 0.1 micrometres to 1.0 micrometres, a thickness in the range 0.3 micrometres to 0.6 micrometres being particularly preferred and a value of substantially 0.4 micrometres being preferred still further. Optionally, the amorphous carbon layer is formed by sputter deposition. Preferably, the sputter deposition is performed from a substantially pure graphite target that may optionally be performed at a pressure of  $1 \times 10^{-6}$  Torr or less. Preferably, the sputtering is performed using an inert gas such as argon. These measures help to maintain the purity of the amorphous carbon. Emissivity has been found to decrease as the level of impurities in the amorphous carbon film increases.

Optionally, a titanium layer is formed as the metallic carbide layer and the titanium layer may optionally be formed by sputter deposition or evaporation. Preferably, the titanium layer is formed to a thickness in the range



- 4 -

of 0.1 micrometres to 1.0 micrometres, a thickness of substantially 0.3 micrometres being particularly preferred.

The method may optionally further comprise the step of forming a protective layer on top of the amorphous carbon layer. Preferably, a layer of  
5 SiC, SiO<sub>2</sub> diamond or diamond-like carbon is formed as the protective layer.

Optionally, the radiator is annealed after the steps of forming the metallic carbide-forming layer and the amorphous carbon layer. Preferably, the radiator is annealed at a temperature in the range 600°C to 900°C, at a temperature of substantially 800°C being particularly preferred. In a currently preferred  
10 embodiment, the radiator is annealed by holding the radiator at a temperature of substantially 800°C for substantially five minutes and may, optionally, be annealed in an inert atmosphere (in argon, for example).

The invention will now be described, by way of example only, by reference to the accompanying drawings in which:

15 Figure 1 is a perspective view of a radiator according to an embodiment of the present invention;

Figure 2 is a simplified representation of apparatus for making a radiator according to a preferred method of the present invention; and

Figure 3 is a graph showing emissivities achieved for a number of  
20 radiators according to an embodiment of the present invention as a function of the thickness of the amorphous carbon film.

A radiator 10 according to an embodiment of the present invention is shown in Figure 1 and comprises a silicon substrate 12 that provides a substrate surface 12a on which is deposited a titanium film 14, an amorphous  
25 carbon film 16 and a protective silicon carbide (SiC) film 18, each film capping the previous one. The titanium, amorphous carbon and SiC films 14, 16, 18 are thin, each being less than a micrometre thick, whereas the thickness of the substrate 12 is much greater in this instance (of the order of millimetres). However, the substrate may be thinner, even as thin as the titanium,  
30 amorphous carbon and SiC films 14, 16, 18. As will be clear, the thicknesses of

- 5 -

the titanium, amorphous carbon and SiC films 14, 16, 18 relative to the size of the substrate 12 in Figure 1 have been increased for the sake of clarity. This radiator 10 exhibits high thermal emissivity in the infra red range.

Figure 2 shows, as a simplified sketch, apparatus suitable for growing  
5 the titanium, amorphous carbon and SiC films 14, 16, 18 on the substrate 12. The substrate 12 is prepared to produce the substrate surface 12a that is both clean and smooth. The substrate 10 is mounted on a substrate table shown schematically at 20 such that the surface 18 faces a sputtering target 22 and ion source 24. The substrate table 20, sputtering target 22 and ion source 24 are  
10 located within a vacuum chamber shown generally at 26.

In order to make the high-emissivity radiator 10, the prepared substrate 12 is held in the vacuum chamber 26 at  $1 \times 10^{-6}$  Torr and the films 14, 16, 18 are deposited onto the substrate surface 12a at this reduced pressure. All three films 14, 16, 18 are deposited using the well-known technique of sputter  
15 deposition. In this embodiment, the ion source 24 is aimed at one of three interchangeable sputtering targets 22, the targets 22 being made of titanium, pure graphite and SiC. The ion source 24 may be of any standard construction, e.g. a diode or magnetron where both electrodes are preferably water-cooled (not shown). High-purity argon gas is used as the source of ions and is  
20 introduced at a flow rate to increase the pressure within the vacuum chamber to 4 milliTor. The substrate table 20 may be earthed or it may be electrically floating.

Initially, the titanium sputter target is placed in the position indicated at 22 and the ion source 24 activated such that a thin film of titanium 12 is deposited  
25 onto the clean substrate surface 12a to a depth of 0.3 micrometres. The depth may be ascertained in any number of standard ways such as using a quartz micro-balance (not shown). The titanium sputtering target is then replaced by the graphite sputtering target at position 22. Activating the ion source 24 results in an amorphous carbon film 16 being deposited onto the titanium film 14. This  
30 second phase of sputtering continues until an amorphous carbon film 16 with a thickness of 0.5 micrometres is deposited. An amorphous carbon film 16

- 6 -

results that is soft and can easily be scratched with a pair of tweezers or the like: this type of film 16 has been found to produce optimal emissivity.

The radiator 10, now comprising substrate 12, titanium film 14 and amorphous carbon film 16, is then annealed at the vacuum chamber's base pressure of  $1 \times 10^{-6}$  Torr, most of the residual gas within the vacuum chamber 5 26 comprising the inert argon that was introduced for sputtering. This is beneficial as the argon will not react with the amorphous carbon film 16 during the annealing process. The radiator 10 is heated such that its temperature slowly increases to 800°C. The radiator 10 is then held at 800°C for five 10 minutes before being allowed to cool slowly to the ambient temperature. This annealing step produces an amorphous carbon film 16 with improved homogeneity and this in turn provides a higher thermal emissivity that is uniform across the radiator 10.

Finally, the graphite target 22 is replaced with the SiC target such that 15 the protective SiC film 18 can be formed on the amorphous carbon film 16 by sputter deposition. The SiC film 18 is deposited to a thickness of between a micrometer or less up to over a millimetre thick, a thickness of 0.1 micrometer being currently preferred. Such a SiC film 18 is transparent to infra red radiation.

20 In this way, a radiator 10 is formed that has high emissivity at wavelengths between 3 and 5 micrometres (in the infra red range). Emissivities as high as 95% have been measured. We have found that the emissivity varies as a function of thickness of the amorphous carbon film 16 as is shown in Figure 3.

25 It will be readily apparent to those skilled in the art that variations to the above described embodiment are possible without departing from the scope of the invention defined in the appended claims.

For example, the films 14, 16, 18 need not entirely cover the substrate 12. The films may cover one or more areas of the substrate surface 12a and 30 may even be formed according to a desired pattern. Where sputtering is used as the method of deposition, masks can be used to achieve the desired pattern.

- 7 -

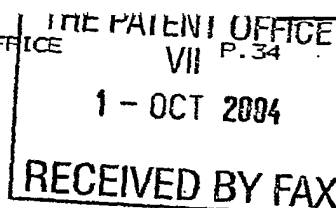
Moreover, the films 14, 16, 18 need not extend to cover the same areas. It is highly advantageous for the titanium and amorphous carbon films 14, 16 to be coterminous, but a protective film 18 may only be needed in selective areas that are particularly prone to damage.

5        Silicon is but one example of a substrate. Essentially, any material may form the substrate if it will allow deposition of titanium and will withstand the annealing process. Suitable substrates include semiconductors and metals.

10        Whilst the above embodiment employs interchangeable sputtering targets 22, the sputtering targets 22 may be located in fixed but different positions. Such sputtering targets 22 may share a common ion source 24 that may be directed to each sputtering target 22 or there may be a dedicated ion source 22 for each sputtering target 22. In addition, argon is but a preferred source of ions. Alternatives, such as nitrogen or oxygen, are also possible.

15        Ion source sputtering is one of many well-known techniques suitable for forming the films 14, 16, 18 on the substrate 12. For example, plasma-source sputtering, chemical vapour deposition, laser ablation, cathodic arc processes or evaporation such as molecular beam epitaxy (MBE) using e.g. thermal or e-beam heat sources may be used to form the films 14, 16, 18.

20        Whilst the radiator 10 has been annealed prior to deposition of the protective SiC film 18, the annealing could equally well be performed after deposition of the SiC film 18.



- 8 -

### CLAIMS

1. A radiator comprising a substrate, an amorphous carbon layer and a metallic carbide-forming layer interposed between the substrate and amorphous carbon layer.
- 5 2. The radiator of claim 1, wherein the metallic carbide-forming layer comprises titanium.
3. The radiator of claim 1 or claim 2, wherein the amorphous carbon layer and/or the titanium layer has a thickness in the range of 0.1 micrometres to 1.0 micrometres.
- 10 4. The radiator of any preceding claim, wherein the amorphous carbon layer is protected by a protective layer.
- 15 5. The radiator of claim 4, wherein the protective layer is substantially transparent to infrared radiation.
6. The radiator of claim 5, wherein the protective layer comprises at least one of SiC, SiO<sub>2</sub>, diamond and diamond-like carbon.
- 20 7. A method of making a radiator comprising the steps of forming a metallic carbide-forming layer on a substrate and forming an amorphous carbon layer on the metallic carbide-forming layer.
- 25 8. The method of claim 7, wherein the amorphous carbon layer and/or the metallic carbide forming layer is formed by sputter deposition or evaporation.

- 9 -

9. The method of claim 7 or 8, further comprising the step of forming a protective layer on top of the amorphous carbon layer.
- 5 10. The method of any of claims 7 to 9, wherein the radiator is annealed after the steps of forming the metallic carbide-forming and amorphous carbon layers.

THE PATENT OFFICE  
VII  
1 - OCT 2004  
RECEIVED BY FAX

- 10 -

**ABSTRACT**

**HIGH-EMISSION RADIATOR**

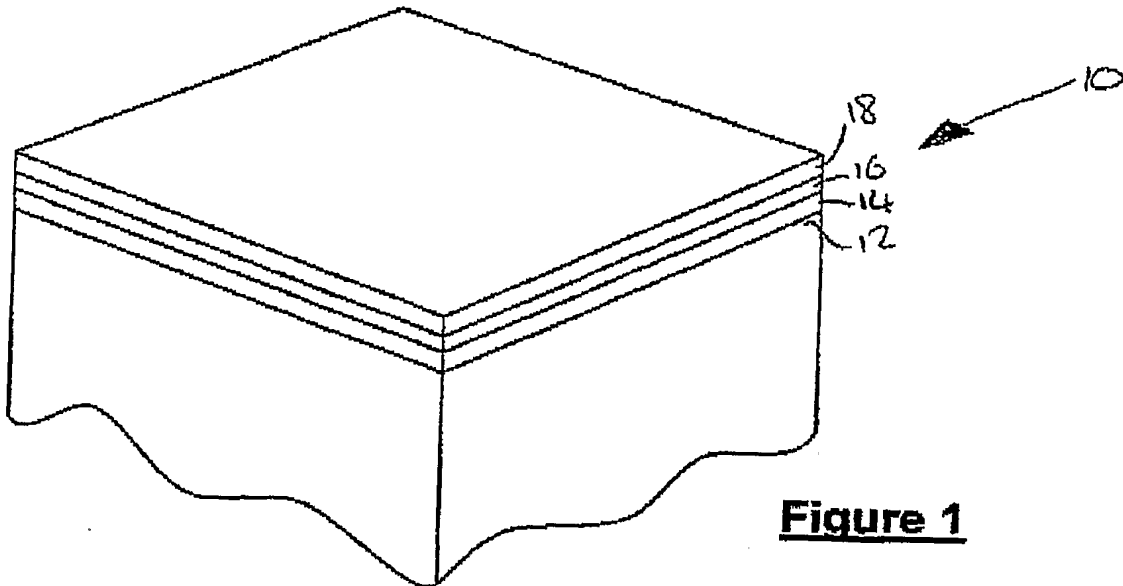
This invention relates to a radiator 10 and to a method of making a  
5 radiator. In particular, this invention relates to a radiator 10 having thin-film  
coatings that serve to increase the thermal emissivity of the entire structure. A  
radiator 10 is provided comprising a substrate 12, an amorphous carbon layer  
16 and the metallic carbide forming layer 14 interposed between the substrate  
12 and amorphous carbon layer 16. In addition, a method of making a radiator  
10 is provided comprising the steps of forming the metallic carbide-forming layer on  
a substrate and forming an amorphous carbon layer on the metallic carbide-  
forming layer.

15 Fig. 1

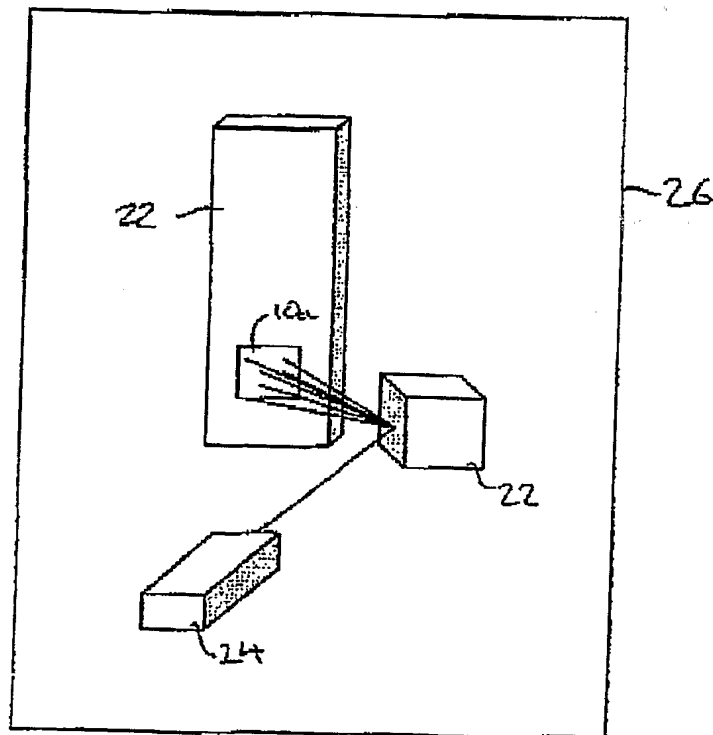
1 - OCT 2004

RECEIVED BY FA

1/2



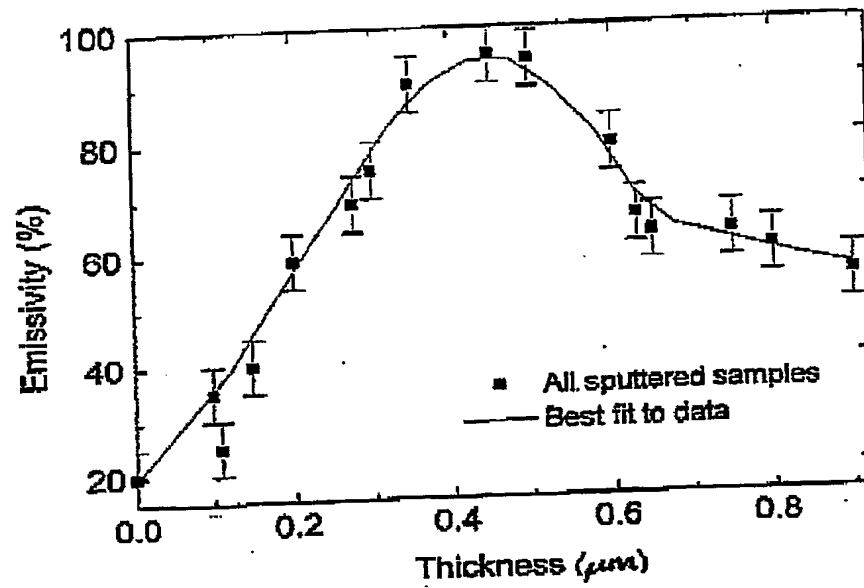
**Figure 1**



**Figure 2**



2/2

**Figure 3**

# PATENT COOPERATION TREATY

From the RECEIVING OFFICE

To:

13

## PCT

NOTIFICATION CONCERNING  
DOCUMENTS TRANSMITTED

Date of mailing  
(day/month/year)

16.11.2005

The receiving Office transmits herewith the following documents:

(number)

1. ☐ \_\_\_\_\_ record copies (Article 12(1))
2. ☐ \_\_\_\_\_ search copies (Article 12(1))
3. ☐ \_\_\_\_\_ translations of international applications (Rule 12.3 or 12.4)
4. ☐ \_\_\_\_\_ copies of purported international applications (Rule 20.7(iv))
5. ☐ \_\_\_\_\_ record copies and corrections not already transmitted in respect of the international applications which have been considered withdrawn (Rule 29.1(a)(i))
6. ☐ \_\_\_\_\_ (copies of the) letters of corrections or rectifications (Administrative Instructions, Section 325(b) and (c))
7. ☒ \_\_\_\_\_ (copies of) replacement sheets (Administrative Instructions, Section 325(b) and (c))
8. ☐ \_\_\_\_\_ (copies of) later submitted sheets (Administrative Instructions, Section 309(b)(iii), (c)(ii))
9. ☐ \_\_\_\_\_ (copies of) later submitted drawings (Administrative Instructions, Section 310(c)(iii), (d)(ii))
10. ☐ \_\_\_\_\_ other documents (specify):

✓	POA
✓	DRAWINGS
	RO/101
	OTHER

☐ The Annex contains a list identifying each document transmitted by the type of document it is, the corresponding international application number and, if necessary, other information.

PCT/GB 2005 / 0 5 0 1 7 1 BAE Systems

This notification is sent to the addressee in its capacity as:

- ☐ the International Searching Authority
- ☒ the International Bureau

Name and mailing address of the receiving Office

The Patent Office  
Cardiff Road, Newport

Facsimile South Wales NP10 800

Authorized officer

PHIL TREEN  
ROOM: GY18

Telephone No.

EXT: 4035



Fig.1.

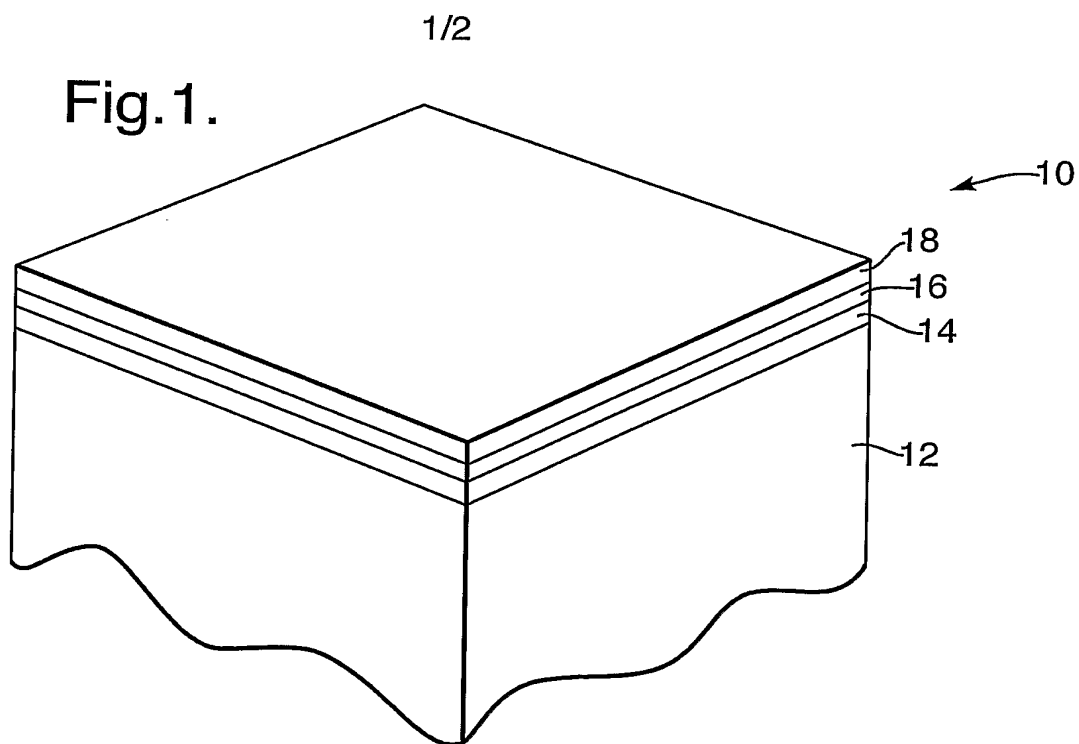
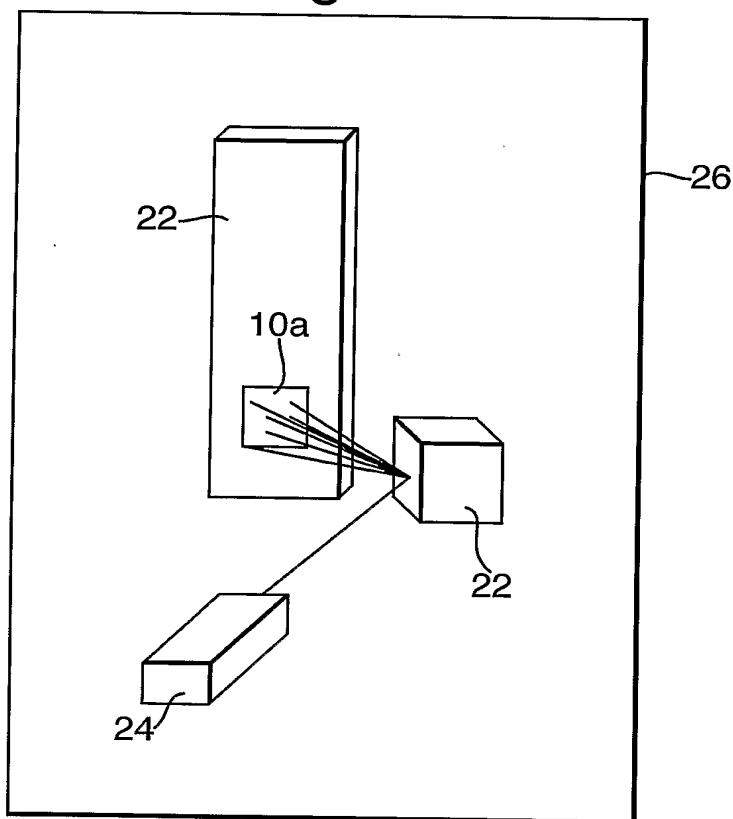


Fig.2.





09 NOV 2005

2/2

Fig.3.

